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Developing an Optimized Process Strategy for Acid Cleaning of the Savannah River Site HLW Tanks

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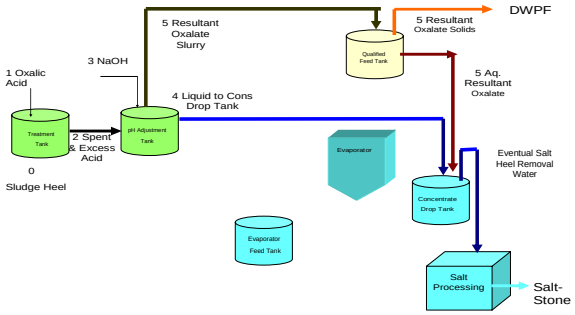
INTRODUCTION

At the Savannah River Site (SRS), there remains approximately 35 million gallons of High Level Waste (HLW) that was mostly created from Purex and SRS H-Area Modified (HM) nuclear fuel cycles. The waste is contained in approximately forty-nine tanks fabricated from commercially available carbon steel. In order to minimize general corrosion, the waste is maintained as very-alkaline solution[1]. The very-alkaline chemistry has caused hydrated metal oxides to precipitate and form a sludge heel[3]. Over the years, the sludge waste has aged, with some forming a hardened crust. To aid in the removal of the sludge heels from select tanks for closure[2] the use of oxalic acid to dissolve the sludge is being investigated.

Developing an optimized process strategy based on laboratory analyses would be prohibitively costly. This research, therefore, demonstrates that a chemical equilibrium based software program can be used to develop an optimized process strategy for oxalic acid cleaning of the HLW tanks based on estimating resultant chemistries, minimizing resultant oxalates sent to the evaporator, and minimizing resultant solids sent to the Defense Waste Processing Facility (DWPF).

PROPOSED FLOWPATH

Figure-1 shows the proposed flowpath.



Figure–1 Proposed Flowpath

SOFTWARE

OLI Stream Analyzer© is the chemical equilibrium-based software that was chosen for this effort. The preference is based on an extensive solubility databank for very alkaline chemistries, its functionality in modeling SRS HLW, and technical support. Before the software is used to estimate resultant chemistries and evaluate the process strategy; a simple test is used to compare the software dissolution forecasts to well documented SRNL dissolutions[4,5]. Since weight percent (wt%) dissolved and precipitate mass are based on the same solubility databank, it is assumed that similarities between the software forecasts and the SRNL results, are also indicative of the software's ability to estimate precipitate mass. Table-1 summarizes the sludge characterizations, while Figure-2 shows the acceptable similarities between the forecasts and the SRNL dissolutions.

Table-1 Chemical Characterization of Sludge[1]

Solid	Purex Sludge Simulant (mole)	HM Sludge Simulant (mole)	Tank 8 Purex[1] Sludge (mole)	Tank 16 HM[1] Sludge (mole)
$Al(OH)_3$	2.2E-1	1.5E+0	3.6E-1	6.9E-01
$Ca_3(PO_4)_2$	1.5E-3	4.8E-5	6.7E-3	NR[2]
$Fe(OH)_3$	8.4E-1	1.8E-1	2.0E+0	1.8E+0
$Mg(OH)_2$	2.6E-2	1.2E-2	NR[2]	NR[2]
$Mn(OH)_2$	4.7E-1	1.1E-1	2.6E-1	3.3E-1
$Mn_3(PO_4)_2$	1.1E-2	1.1E-2	6.1E-3	1.1E-2
HgO	1.9E-03	2.6E-02	3.0E-3	6.5E-2
$Ni(OH)_2$	9.0E-01	1.6E-02	2.0E+0	6.5E-2

Notes:
1. Sludge has been subsequently removed from the tank, and data, therefore, does not reflect the current tank status.
2. NR denotes “not reported.”

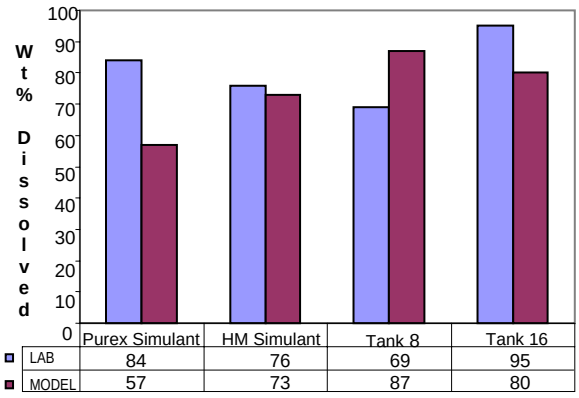


Figure-2 Forecasts vs. SRNL Results

As part of forecasting resultant chemistry and assessing the process strategy, the following operations are modeled using the software.

- Oxalic acid (Stream #1), based on an assumed 100% molar excess required to drive the chemical reaction towards dissolution, is added to the Treatment Tank to dissolve an assumed 5,000-gal of either Purex sludge or HM sludge (Stream #0).

- The pH Restoration Tank is pre-charged with enough sodium hydroxide (Stream #2) to adequately restore the pH of the spent/dissolved acid solution to within the corrosion control program.
- After allowing adequate time, as well as performing adequate mixing, the spent excess acid/dissolved solids (Stream #2) are transferred to the pre-treated pH Restoration Tank.
- Sodium hydroxide in the pH Restoration Tank causes solids to precipitate, resulting in a liquid and a solids phase (total added volume = Stream #4 + Stream #5).
- Assuming that precipitated solids are transferred as 16-wt% solids slurry, the resulting decanted liquid fraction from Stream #5, will be eventually transferred to the Condensate Drop Tank.
- After the slurry (Stream #5) is added to a qualified sludge batch, it is provided adequate time to allow for settling. The liquid decant fraction of Stream #5 will then be transferred to the Condensate Drop Tank.

For the treatment of the assumed Purex sludge heel (Tank 8) and assumed HM sludge heel (Tank 11), the Stream Analyzer© calculated material balance is shown in Table-2 and Table-3, respectively.

Table-2 Generated Material Balance for Tank 8 Purex Sludge

Stream	Stream #0 **Sludge[1] Heel**		Stream #1 **Oxalic Acid**	Stream #2 **Spent / Excess Acid**		Stream #3 **50 wt% NaOH**	Stream #4 **Liquid to Drop Tank**	Stream #5 **Resultant Slurry to DWPF**	
Phase	**Aq[2]**	**Solid**	**Aq[2]**	**Aq[2]**	**Solid**	**Aq[2]**	**Aq[2]**	**Aq[2]**	**Solid**
Units	**Weight Fraction**								
H_2O	9.3E-1	0	9.2E-1	9.1E-1	0	0	9.6E-1	9.6E-1	9.6E-1
$H_2C_2O_4$	0	0	8.0E-2	4.0E-2	0	0	0	0	0
HCl	0	0	0	4.6E-4	0	0	0	0	0
HNO_3	0	0	0	4.8E-4	0	0	0	0	0
Na_2CO_3	3.4E-4	0	0	2.1E-3	0	0	2.1E-3	2.1E-3	0
NaCl	1.2E-2	0	0	0	0	0	7.1E-4	7.1E-4	0
$NaNO_2$	0	0	0	0	0	0	0	0	0
$NaNO_3$	1.1E-2	0	0	0	0	0	6.2E-4	6.2E-4	0
NaOH	3.4E-2	0	0	0	0	5.0E-1	4.6E-3	4.6E-3	0
Na_2SO_4	0	0	0	0	0	0	0	0	0
$Na_2C_2O_4$	0	0	0	4.4E-3	0	0	2.9E-2	2.9E-2	7.0E-1
$Al(OH)_3$	0	1.5E-1	0	0	0	0	0	0	4.2E-2
AlOOH	0	0	0	4.0E-3	0	0	0	0	0
$NaAlO_2$	3.3E-3	0	0	0	0	0	6.6E-4	6.6E-4	0
CaC_2O_4	0	0	0	4.1E-4	0	0	7.6E-7	7.6E-7	2.4E-2
$CaCO_3$	0	6.2E-2	0	0	2.8E-1	0	0	0	0
$Ca(OH)_2$	2.4E-4	0	0	0	0	0	0	0	0
Ce_2O_3	1.3E-3	0	0	0	0	0	7.6E-5	7.6E-5	0
$Ce_2(C_2O_4)$	0	0	0	3.8E-5	9.1E-3	0	0	0	0
$Fe_2(C_2O_4)$	0	0	0	3.3E-2	0	0	2.6E-5	2.6E-5	0
$Fe(OH)_3$	7.4E-5	5.8E-1	0	0	0	0	0	0	1.7E-1
$K_2C_2O_4$	0	0	0	0	0	0	0	0	0
KOH	0	0	0	0	0	0	0	0	0
MnC_2O_4	0	0	0	4.5E-4	3.4E-1	0	2.8E-6	2.8E-6	0
$Mn(OH)_2$	8.8E-7	6.0E-2	0	0	0	0	0	0	1.8E-2
NiC_2O_4	0	0	0	5.7E-5	2.9E-1	0	9.5E-8	9.5E-8	0
$NiOH_2$	3.1E-7	4.5E-2	0	0	0	0	0	0	1.4E-2
SiO_2	1.2E-2	0	0	3.8E-3	8.0E-2	0	7.1E-4	7.1E-4	0
$UO_2C_2O_4$	0	0	0	3.8E-3	0	0	1.1E-4	1.1E-4	0
UO_2OH_2	2.7E-8	9.9E-2	0	0	0	0	0	0	2.9E-2
Total kg[3]	1.7E+4	9.4E+3	2.6E+5	2.9E+5	2.6E+3	3.9E+4	1.4E+5	1.6E+5	3.2E+4

Notes:

1. Sludge has been subsequently removed from the tank and data, therefore, does not reflect the current tank status.
2. "Aq." denotes aqueous.
3. Total kg. may not equal 100% because of rounding.

Table-3 Generated Material Balance for Tank 11 HM Sludge Dissolution

Stream	Stream #0 Sludge[1] Heel		Stream #1 Oxalic Acid	Stream #2 Spent Excess Acid		Stream #3 50 wt% NaOH	Stream #4 Liquid to Drop Tank	Stream #5 Resultant Slurry to DWPF	
Phase	**Aq[2]**	**Solid**	**Aq[2]**	**Aq[2]**	**Solid**	**Aq[2]**	**Aq[2]**	**Aq[2]**	**Solid**
Units	**Weight fraction**								
H_2O	9.4E-1	0	9.2E-1	9.1E-1	0	5.0E-1	9.6E-1	9.6E-1	0
$H_2C_2O_4$	0	0	8.0E-2	6.3E-2	0	0	0	0	0
HCl	0	0	0	8.7E-5	0	0	0	0	0
HNO_3	0	0	0	5.1E-4	0	0	0	0	0
Na_2CO_3	6.2E-5	0	0	4.4E-4	0	0	4.3E-4	4.3E-4	0
NaCl	3.1E-3	0	0	0	0	0	1.3E-4	1.3E-4	0
$NaNO_2$	0	0	0	0	0	0	0	0	0
$NaNO_3$	1.6E-2	0	0	0	0	0	6.7E-4	6.7E-4	0
NaOH	1.8E-2	0	0	0	0	5.0E-1	4.4E-3	4.4E-3	0
Na_2SO_4	0	0	0	0	0	0	0	0	0
$Na_2C_2O_4$	0	0	0	1.8E-3	0	0	3.1E-2	3.1E-2	7.7E-1
$Al(OH)_3$	0	5.9E-1	0	0	0	0	0	0	4.2E-2
AlOOH	0	0	0	1.1E-2	0	0	0	0	0
$NaAlO_2$	4.4E-5	0	0	0	0	0	6.1E-4	6.1E-4	0
CaC_2O_4	4.9E-5	0	0	4.0E-5	0	0	7.4E-7	7.4E-7	2.4E-2
$CaCO_3$	0	1.7E-2	0	0	0	0	0	0	0
$Ca(OH)_2$	4.3E-5	0	0	0	0	0	0	0	0
Ce_2O_3	2.7E-3	0	0	0	0	0	1.2E-4	1.2E-4	0
$Ce_2(C_2O_4)_3$	0	0	0	6.1E-5	0	0	0	0	0
$Fe_2(C_2O_4)_3$	0	0	0	1.1E-2	0	0	2.4E-5	2.4E-5	0
$Fe(OH)_3$	9.6E-7	2.5E-1	0	0	0	0	0	0	6.2E-2
$K_2C_2O_4$	0	0	0	0	0	0	0	0	0
KOH	0	0	0	0	0	0	0	0	0
MnC_2O_4	0	0	0	2.8E-4	0	0	3.6E-6	3.6E-6	0
$Mn(OH)_2$	1.0E-7	5.9E-2	0	0	0	0	0	0	1.42E-2
NiC_2O_4	0	0	0	5.1E-5	3.3E-1	0	8.9E-8	8.9E-8	0
$Ni(OH)_2$	4.5E-9	1.1E-2	0	0	0	0	0	0	2.7E-3
SiO_2	2.0E-2	0	0	1.0E-4	6.7E-1	0	8.7E-4	8.7E-4	0
$UO_2C_2O_4$	0	0	0	9.0E-4	0	0	1.2E-6	1.2E-6	0
UO_2OH_2	2.7E-7	3.1E-2	0	0	0	0	0	0	7.5E-3
ThO_2	4.9E-14	1.1E-2	0	0	0	0	4.8E-14	4.8E-14	2.7E-3
HgO	3.9E-5	3.1E-2	0	7.6E-4	0	0	3.8E-5	3.8E-5	7.1E-3
$Th(C_2O_4)_2$	0	0	0	4.3E-4	0	0	0	0	0
Total kg[3]	1.6E+4	9.4E+3	3.5E+5	3.7E+5	1.8E+3	5.2E+4	1.9E+5	1.9E+5	3.8E+4

Notes:
1. Sludge has been subsequently removed from the tank and data, therefore, does not reflect the current tank status.
2. “Aq” denotes aqueous.
3. Total kg may not equal 100% because of rounding.

Precipitation in the Evaporator

The disposition options for Stream #4 and the aqueous decant of Stream #5 are determined using a model similar to a 242-16H evaporator model[6] and assuming recent Evaporator Feed Tank compositions. Refer to Table-4.

Table-4 Evaporator Feed Tank Initial Composition

Chemical Compound	Feed Tank (mole)
$NaNO_3$	1.82
$Na_2CO_3.H_2O$	0.0876
$NaNO_2$	1.69
$NaAlO_2 \cdot 2H_2O$	0.114
$Na_2C_2O_4$	0.00619
Na_2SO_4	0.0239
NaCl	0.00348
NaF	0.00579
NaOH	4.71
Na_3PO_4	0.00651
Na_2SiO_3	0.00784

Using Table-4, Stream #4, a sodium oxalate balance is constructed across the evaporator system (i.e., starting at the Evaporator Feed Tank, going through the evaporator, and including the Condensate Drop Tank). As seen in Table-5, if the soluble oxalates of Stream #4 are added to the Evaporator Feed Tank, approximately 20% to 30% of the sodium oxalate would remain soluble, flow through the evaporator, and precipitate in the Condensate Drop Tank. Based on past operating experience, a flow of 20% to 30% of this oxalate through the evaporator would result in an increased need for evaporator pot de-scaling, while also decreasing the evaporator space attainment. Additionally, if Stream #4 is added to the Condensate Drop Tank, the sodium concentration would cause almost all of the soluble oxalate to precipitate.

Table-5 Sodium Oxalate Balance Across Evaporator

Tank	Basis	Tank 8		Tank 11	
		$Na_2C_2O_4$ (kg)	% of added	$Na_2C_2O_4$ (kg)	% of added
Initial Mass in Feed Tank	Ref. Hang, 2002	2,940	-	2,670	-
Added to Feed Tank	Table 2, Stream #4 Table 3, Stream #4	4,089 -	- -	- 5,901	- -
Remains in Feed Tank	Model Output	5,131	73%	6,942	81%
Processed through Evaporator to Condensate	Model Output	1,968	28%	1,629	19%

During the routine recycling of small quantities of liquid from the Condensate Drop Tank back to the Evaporator Feed Tank, the low soluble sodium oxalate concentration would be diluted with existing supernate in the Evaporator Feed Tank such that significant precipitation should not occur until the oxalate is back in the Condensate Drop Tank. The assumed flowpath for Stream #4, and the decanted aqueous fraction of Stream #5, will transfer the liquid to the Condensate Drop Tank. The Condensate Drop Tank's high sodium concentration will cause the soluble oxalate to quickly precipitate and form part of a salt heel. The salt heel will safely remain in the Condensate Drop Tank, until at some future time, when as part of closure, a salt heel sodium oxalate removal program is initiated.

REMOVAL OF THE OXALATE HEEL

To better quantify the resultant oxalate impact, we consider the effect of dissolving the remaining sodium oxalate heel from an initial 1,000,000-gal of saltstone. With a sodium concentration of 6.4 [M], the maximum sodium oxalate solubility is shown by Equation-1[7] .

$$\text{Maximum}[Na_2C_2O_4] = T \cdot \left(0.00159 \cdot I^{-1.444}\right) + \left(0.0723 \cdot I^{-1.424}\right) \qquad \text{(Eq-1)}$$

where:

T = temperature in Celsius

I = total sodium concentration [M]

Assuming 30°C, the sodium oxalate is saturated at 0.0084 [M]. For one million gallons of saltcake, the soluble sodium oxalate is calculated to be about 11,800-kg. If the saltcake contains more sodium oxalate, it precipitates in the low solubility heel. Based on sampling, average SRS saltcake solids contain about 0.45-wt% sodium oxalate[7]. In one million gallons of saltcake, with a solid void fraction of 0.40, and a solids specific gravity of 2.3, saltcake contains about 23,500-kg of sodium oxalate.

This means that during the dissolution of each tank filled with saltcake, out of the 23,500-kg, about 11,750-kg will be soluble and about 11,750-kg will form an insoluble heel. With the maximum sodium oxalate saturation equal to 0.0084 [M], about 2,800,000-gal of water will eventually be needed to dissolve the heel as part of a formal salt heel removal effort.

Table-3 and Table-4 show that Stream #4 will increase the sodium oxalate by about 4,089-kg for Purex sludge and 5,901-kg for HM sludge. The decanted aqueous fraction of Stream #5 will also increase the sodium oxalate by 4,640-kg for Purex sludge and 5,901-kg for HM sludge. By adding the two together, the total sodium oxalate solids in the Condensate Drop Tank from the dissolution of 5,000-gal of Purex sludge will be 8729-kg, while for 500-gal of HM sludge the total oxalate solids will be 11,800-kg. For each 5,000-gal of either Purex sludge or HM sludge treated, less than 2,000,000-gal or 2,800,000-gal of water will eventually be required to dissolve the salt oxalate heel, respectively.

By comparing the oxalate solids from the dissolution of 1,000,000-gal of saltcake to the resultant oxalate heel from oxalic acid cleaning of 5,000-gal of sludge, we see that comparable quantities of sodium oxalates solids are created. Evident also is the fact that comparable quantities of water will ultimately be required to dissolve the oxalate heels as part of a salt heel dissolution effort.

IMPACT ON DWPF

If the Stream #5 solids are washed as part of feed qualification, nearly all of the initially precipitated sodium oxalate will dissolve and eventually precipitate in the evaporator system. Stream #5, therefore, should be added directly to a pre-washed qualified sludge batch. After being added to the qualified feed tank, settling time should be allowed such that the liquid fraction of Stream #5 can be decanted back to the Condensate Drop Tank. Based on a review of DWPF processing, the possible impacts of sodium oxalate solids on vitrification can be divided into the following three types.

1) Impacts on the sludge receipt and adjustment tank (SRAT).
2) Impacts on melter behavior

3) Impacts on glass performance

Previously, as part of qualifying Sludge Batch #3, the effects of substantial concentrations of sodium oxalate in the sludge were evaluated. Table-6 shows the sodium oxalate content in the feeds tested [8].

Table-6 Sodium Oxalate Content for Sludge Batch#3 Testing

wt% Sodium Oxalate in Total Solids	% Washed Out	% Remaining
	Relative to Total Sodium Oxalate in Source Tank	
14%	75	25
26%	50	50
34%	25	75
46%	-25	125

The studies[8] showed that the DWPF SRAT is affected as follows:

- If sodium oxalate content is kept below 26-wt%, the solubility of iron and gadolinium are not affected, but when the sodium oxalate increases to 34-wt%, iron becomes 10x more soluble, while gadolinium becomes completely soluble.

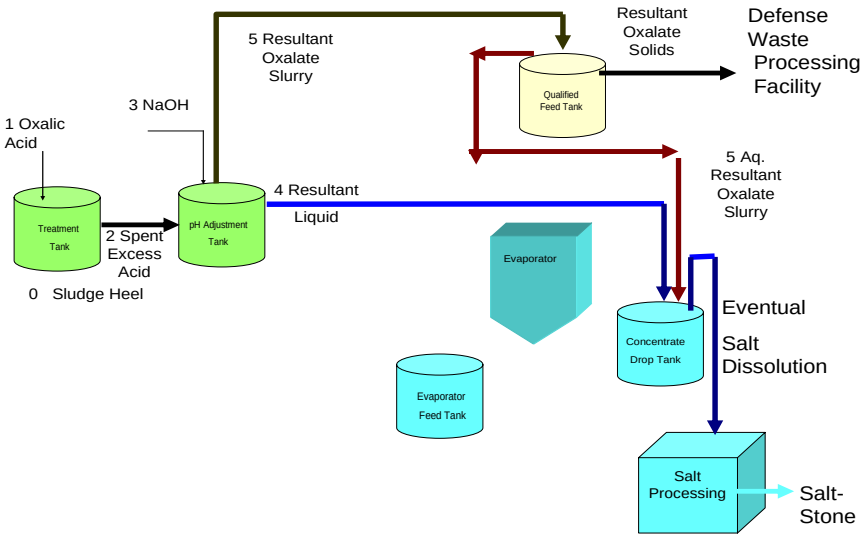
Preliminary studies to determine melter behavior and glass acceptability of the elevated sodium oxalate planned for Sludge Batch #3 shows mixed results. Frit variation studies show that acceptable glass can be made, and the higher waste loadings are acceptable; however, DWPF processing issues, such as melt rate, constrain waste loading such that maximum loadings are difficult to achieve. Current processing of Sludge Batch #3 demonstrates that acceptable glass can be made with a total sodium content of the composite frit-sludge mixture greater than about 1.0 [M] [9]. Projecting that a similar total oxalate content can be made acceptable in future batches, the sodium contributed from the frit can be decreased to allow for an increase from Stream #5. In addition, considerably more sodium oxalate could be added, if more sodium is first removed by further washing the sludge, prior to adding the oxalate slurry.

Adjusting frit and further washing the sludge can compensate for an increased sodium oxalate concentration; therefore, an increase in DWPF canisters (relative to sludge processed) is not necessary. Given the experience with Sludge Batch #3, considerable sodium oxalate could be added with negligible difference to the process or canisters produced, as long as the oxalate addition is considered in batch planning and qualification testing. Although various changes can be made to allow an increased sodium oxalate concentration in the sludge, given that DWPF can only readily accommodate only a small sodium oxalate concentration (about 10-wt%), the preferred option must remain to add the slurry to a pre-washed qualified DWPF feed sludge in a slow steady state (i.e., the solid oxalates from about two or three 5,000-gal of sludge heel treatments can be added to one sludge batch)

.

CONCLUSION

This research has shown the use of a chemical equilibrium based software to optimize the processing strategy for the oxalic acid treatment of a sludge heel. Since the oxalate impact to the evaporator system and DWPF are already minimized with the current assumed flowpath, as shown in Figure-3, no changes to either the Stream #4 or the Stream #5 processing strategies are needed.



Sludge	Stream #0	Stream #1	Stream #2		Stream #3	Stream #4	Stream #5		**Event. Water to Diss. Oxalate Heel**
	Sludge Heel (gal)	Oxalic Acid (gal)	Aq. Spent Acid/ Diss. Solids (kg)	Sol. Spent Acid/ Diss. Solids (kg)	50% wt NaOH Sol (gal)	Liquid to Cond. Drop Tank (kg)	Aq. to Qual Feed (kg)	Solids to Qual Feed (kg)	**(gal)**
Tank 8 Purex	5.0E+3	6.5E+4	2.9E+5	2.6E+3	6.8E+3	1.4E+5	1.6E+5	3.3E+4	<2.0E+6
Tank 11 HM	5.0E+3	8.5E+4	8.9E+4	8.2E+1	9.0E+3	1.9E+5	1.9E+5	3.8E+4	<2.8E+6

Figure-3 Summary of Recommended Flowsheet

For the oxalic acid dissolution of a 5,000-gal sludge heel, the soluble sodium oxalate will be sent eventually to the Condensate Drop Tank. In the Condensate Drop Tank, the sodium oxalate will quickly precipitate insoluble oxalate heel. This insoluble heel should only be removed as part of a formal salt heel removal effort. For comparative purposes, the oxalate heel from the oxalic acid dissolution of 5,000-gal of sludge is comparable to the sodium oxalate heel resulting from the dissolution of one HLW tank full of saltcake.

Although various changes can be made to allow an increased sodium oxalate concentration in the sludge, given that DWPF can readily accommodate only a small sodium oxalate concentration (about 10-wt%), the preferred option is to add the solids at a slow steady rate (i.e., the solid oxalates from about two or three 5,000-gal of sludge heel treatments can be added to one sludge batch) to ensure that the need for additional canisters is minimized. For other Purex and HM sludge dissolutions, the flowsheet values may vary somewhat depending on the actual tank's characterization, compositional variations, and chosen molar excess for both the acid and caustic.

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